

Appl. No. : **Unassigned**
Filed : **Herewith**

AMENDMENTS TO THE SPECIFICATION

Kindly amend the paragraph which beings on page 1, line 17, as follows:

There is an increasing interest in nano- and micron sized materials in numerous technical applications. Such nanostructured materials in the form of nanocrystalline films and powders are cornerstones in the attempt to develop and exploit nanotechnology, they ~~exhibits~~ exhibit properties, which are significantly different from those of the same materials of larger size. During the last decade the insight into nanostructured materials ~~have~~ has dramatically improved through the application of new experimental methods for characterization of materials on the nanoscale. This has resulted in the synthesis of unique new materials with unprecedented properties. For nanostructured coatings, physical properties such as elastic modulus, strength, hardness, ductility, diffusivity and thermal expansion coefficient can be manipulated based on nanometer control of the primary particle or grain size. For nanostructured powders, parameters such as the surface area, solubility, electronic structure and thermal conductivity are uniquely size dependent.

Kindly amend the paragraph which beings on page 2, line 22, as follows:

Wet chemistry synthesis methods such as chemical precipitation, hydrothermal and sol-gel synthesis are the major low temperature processes for production of nanomaterials with nano-scaled primary particles or grains. Such basic synthesis techniques ~~is~~ are based on chemical precipitation of particles from chemical solutions e.g. by creation of a new phase in a chemical reaction or by super saturation of a soluble phase.

Kindly amend the paragraph which beings on page 3, line 19, as follows:

The key drawbacks from the sol-gel process are that it is time consuming, and need after treatment such as drying and calcinations. In the conventional sol-gel process it is necessary to calcine the product for up to 24 hours in order to obtain a crystalline product. In a addition to a higher energy usage and more complicated process it has the unfortunate effect that substantial growth of primary particles occur, and that the specific surface area may decrease by up to 80 %.

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Kindly amend the paragraph which beings on page 4, line 14, as follows:

Though many CO₂ applications have been developed or are under development, high pressure CO₂ also exhibits some limitations. Since CO₂ is non-polar and has weak Van der Waal forces, both polar and non-polar non-volatile molecules often exhibits limited solubility or are virtually insoluble. For example, insoluble compounds such as electrolytes, bio molecules, polymers and inorganic compounds can not be directly processed in high pressure CO₂.

Kindly amend the paragraph which beings on page 5, line 15, as follows:

Other techniques include Precipitation from Gas-Saturated Solutions (PGSS), which involves melting the material to be processed, and subsequently dissolving a supercritical fluid under pressure. The saturated solution is then expanded across a nozzle, where the supercritical fluid, which is more volatile, escapes leaving dry fine particles.

Kindly amend the paragraph which beings on page 5, line 20, as follows:

All these techniques have been successfully used in small scale to produce micron sized particles of various materials for numerous applications. Excellent reviews of prior art supercritical particle formation processes can be found in e.g. Ya-Ping Sun("Supercritical Fluid Technology in Materials Science and Engineering – Syntheses, Properties and Applications, Marcel Dekker Inc., 2002-ISBN: 0-8247-0651-X), Gentile et al (WO03/035673A1), Gupta et al (US2002/0000681A1), Mazen et al (EP0706421B1), Del Re et al (WO02/068107A2), Mazen et al (WO99/44733), Calfors et al, Jagannathan et al (WO03/053561), all of which are hereby ~~included~~ incorporated by reference.

Kindly amend the paragraph which beings on page 5, line 30, as follows:

However, all these techniques suffer from some inherent limitations. The RESS technique is limited by the solvent capacity in the supercritical fluid. For example, supercritical carbon dioxide, which is a preferred solvent in many applications, is limited by a low solubility of ~~towards~~ polar substances. Modifiers such as co-solvents and surfactants may be added to the supercritical carbon dioxide to improve the solubility of the material of interest. Drawbacks of the RESS technique includes that isenthalpic expansion over the nozzle results in large

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temperature drops, which can cause freezing of the solid and carbon dioxide and thereby cause blocking of the nozzle. The nozzle design is further critical for the final particle characteristics such as size and morphology etc. All these drawbacks from microscopic variables limit the control over the process itself, and make scale up difficult.

Kindly amend the paragraph which beings on page 6, line 4, as follows:

Due to its higher solubility the SAS technique and its derivatives generally have higher through puts, and it generally produces particles in the range of 1-10 micron (Gupta et al, US2002/0000681A1). The key and particle size controlling step of the SAS techniques is the mass transfer rate of the antisolvent into the droplet. Hence, mixing of solution and the supercritical fluid is crucial in order to obtain an intimate and rapid mixing, a dispersion of solution as small droplets into the supercritical fluid is required. Various nozzle designs have been proposed to inject solution and supercritical fluid into a particle formation vessel in order to provide a good mixing. Recent modifications of the SAS technique to reduce the particle size includes atomisation techniques such as special designed coaxial nozzles, vibrational atomisation, atomisation by high frequency sound waves, ultrasonic atomisation etc. (US20020000681A1). Though these modified techniques are believed to provide enhanced mass transfer and to result in reduced particle sizes, too rapid particle formation may reduce the control of the size and morphology such as crystallinity of the formed particles, be sensitive to the nozzle design and blockages of the nozzle and be difficult to scale-up.

Kindly amend the paragraph which beings on page 6, line 21, as follows:

A more recent development and versatile method for production of fine particles involves the use of micellar structures as templates or nano-reactors. Micellar structures or emulsions are among the most frequently found colloidal systems, largely present in foods, cosmetics, pharmaceuticals, oils processing, paints etc. They are colloidal dispersions of at least two immiscible fluids. The structure of the emulsions consists of droplets of a dispersed phase in a continuous phase. Typically such micelles are formed using surfactants to reduce the interfacial tension and stabilize the micelles. Such surfactants are amphiphilic molecules containing both a hydrophilic and a lipophilic segment. In normal micelles the continuous phase is an aqueous and the

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lipophilic segment is arranged to interact with an organic phase. The opposite structures are called reverse micelles, and may be water-in-oil emulsions or water-in-CO₂ emulsions.

Kindly amend the paragraph which beings on page 6, line 34, as follows:

Such reverse micelles and micro emulsions allow highly polar or polarizable compounds to be dispersed in this non-polar fluid. A wide range of nanomaterials can now be synthesized using ionic species or water soluble compounds as starting materials in the water cores of the micro emulsions. In these micro emulsion systems, the content of different micellar cavities undergo exchange when the micelles collide, creating opportunities for mixing and reactions between the reactants in the in the different cavities. Examples of applications of such systems are given in e.g. KP Johnson et al, "Water-in Carbon Dioxide micro emulsions: an environment of hydrophiles including proteins", Science: 271: 624-626, 1996, M. Ji et al: Synthesizing and dispersing silver nanoparticles in a water in super critical carbon dioxide micro emulsion, J. Am. Chem. Soc. 121: 2631-2632, 1999, H. Ohde et al: Synthesizing silver halide nanoparticles in supercritical carbon dioxide CO₂ utilizing a water-in-CO₂ micro emulsion), hereby ~~included~~ incorporated by reference. Other uses of such micro emulsions can be found in K.A. Barscherer et al, "Micro emulsions in compressible fluids – a review", Phase Equilibria 107:93-150, 1995. The water-in-CO₂ micro emulsions may also be used as a medium for conducting electrochemistry in dense fluids (Ya-Ping Sun, Supercritical Fluid Technology in Materials Science and Engineering – Synthesis, Properties and Applications", Marcel Dekker, Inc., New York 2002, ISBN 0-8247-0651-X). Other applications include protein extraction, biocatalysis, dispersion polymerisation, emulsion polymerisation, metals extraction, dry cleaning, nanoparticle formation.

Kindly amend the paragraph which beings on page 7, line 36, as follows:

However, though tremendous opportunities exist in applying such micro emulsions the practical embodiments for them are still limited and rather inefficient. A water- in- CO₂ emulsion is typically formed batch wise by adding water, proper surfactants, and electrolytes into a stirred pressure vessel, and compressing the vessel to the desired pressure by adding CO₂ and stirring the vessel in a time sufficient for the micro emulsions to form.

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Kindly amend the paragraph which beings on page 8, line 13, as follows:

Summary of the Invention

Hence, an objective of the present invention may be to provide a semi-continuous or continuous method and apparatus for producing micro emulsions of a controllable size.

Kindly amend the paragraph which beings on page 8, line 17, as follows:

Another objective of the present invention may be to provide a methods and measures for improving the solubility of species, which exhibits a low solubility and/or are virtually insoluble in dense phase CO₂, so they can be dispersed and transported in dense phase CO₂.

Kindly amend the paragraph which beings on page 10, line 4, as follows:

In the present description the term near critical is intended to mean a compressed fluid being close to its critical temperature and pressure such as a fluid being maximum 20 bars and maximum 20 °C from its critical point. An example of a preferred near critical fluid according to the present invention is liquid CO₂ at a pressure of 60 bars and 20 °C.

Kindly amend the paragraph which beings on page 10, line 10, as follows:

Many preferred embodiments according to the present invention further comprises withdrawing in at least part time of said method a fluid stream comprising said micro emulsions suspended, dispersed or dissolved in said fluid being in a near critical or supercritical state.

Kindly amend the paragraph which beings on page 10, line 19 as follows:

Compressed CO₂ is a particularly preferred fluid in many applications of the present invention due to its relatively low critical pressure and temperature, readily availability, inexpensiveness and non-toxic nature. Hence, one of the fluids often comprises compressed CO₂ such as CO₂ in a liquid or a supercritical state.

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Kindly amend the paragraph which beings on page 10, line 24, as follows:

Another important fluid is water or a water mixture comprising one or more substances being dissolved or dispersed therein. Said substances may be substantially insoluble in the compressed CO₂ and may comprise polar molecules and/or polarizable molecules and/or non-polar ~~non-~~ non-volatile molecules. Other important fluids in relation to the present invention are organic solvents such as an oil.

Kindly amend the paragraph which beings on page 11, line 9, as follows:

The amount of said surfactant being selected in an embodiment according to the present invention generally depends on the specific application and the desired stability of the micro emulsions for that application. Factors affecting the optimal include operating pressure and temperature for the specific application, the type of surfactant, the type and the activity of the second fluid, the amount and type of substances dissolved and/or dispersed within said second fluid.

Kindly amend the paragraph which beings on page 12, line 30, as follows:

A particular preferred embodiment involves production of two or more micro emulsions of different composition in separate pressurised vessels and combining said fluids containing said micro emulsions in an external device. Said two or more micro emulsions of different composition may preferably be produced using at least two different surfactants. Said surfactants may be designed with electrostatic forces so as to facilitate contact between micelles of different type and to reduce merging of micelles of the same type. Such electrostatic forces may be introduced by including a molecular charge displacement in the lipophilic part of the surfactant(s). Said molecular charge displacement may be obtained by introducing polarity from organic groups selected from halogenated alkyls and/or halogenated aryls and/or aldehydes and/or ketones and/or ethers and/or hetero-cyclic structures containing oxygen, nitrogen and/or sulphur and/or amides and/or mercaptanes.

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Kindly amend the paragraph which beings on page 13, line 5, as follows:

Many applications according to the present invention involve the use of said micro emulsions produced as nanoreactors for the synthesis of materials having nano-sized primary particles. The term primary particles in this context are intended to mean grains, crystallites, etc. Said primary particles may be in an amorphous or crystalline phase or a combination of the two, and may be formed from one or more chemical reactions occurring within said micro emulsions.

Kindly amend the paragraph which beings on page 13, line 12 as follows:

Further the micro emulsions may be used for shaping said primary particles into a specific shape, size and/or structure. In many embodiments the average size of nanoparticle material formed is maximum 5000 nm, such as an average size of maximum 500 nm, preferably the average size is maximum 100 nm, and most preferably the average size is maximum 30 nm, such as maximum 15 nm. In a particularly preferred embodiment the average size of said nano particle material formed is in the range 0,1-30 nm such as in the range 1-10 nm. The average diameter in this context refers to the average diameter of the primary particles, and as said primary particles may have an irregular shape, the average diameter in this context shall preferably be interpreted as an equivalent spherical diameter. Various techniques of varying quality exists for determination of the size of nanoscaled particles. For clarity the average diameters above refers to equivalent spherical diameters determined by Small Angle X-ray Scattering (SAXS) by applying the Beaucage model [G. Beaucage et al, Journal of Noncrystalline Solids 172-174, p.797-805, 1994]. The Beaucage model is fitted to a specific shape of the particles formed and it is recommended that the shape is checked by a suitable microscopic technique such as Transmission Electron Microscopy (TEM) or Scanning Electron Microscopy (SEM) for consistency.

Kindly amend the paragraph which beings on page 15, line 14 as follows:

In a number of important embodiments of the present invention the pressurized vessel is agitated. The agitation may be provided by a motor driven mixer such as an impeller. The rotating speed of said impeller in said pressurized vessel is often ~~relative~~ relatively low such as a rotating speed in the range 100-5000 rpm, such as a rotating speed of said impeller in the range 250-3000 rpm, and preferably in the range 500-2000 rpm.'

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Kindly amend the paragraph which beings on page 16, line 11 as follows:

One preferred embodiment according to the present invention, comprises a plurality of hollow fibre membranes. Typically the hollow tubular members may be arranged into one or more section(s) or array(s) having a porous structure of any shape. Said one or more section(s) may further be arranged within a pressure housing such as a pressure vessel. Various ways of arranging such fibres are known in the prior art (e.g. W.S. Ho et al, "Membrane Handbook", Van Nordstrand Reinhold, 1992, ISBN 0-442-23747-2, K. Scott, "Handbook of Industrial Membranes", Elsevier Publicers, 1995, ISBN 1856172333, Iversen et al, WO95351153, Iversen et al, WO00160095, US690,830, US5,690,823) and are hereby ~~included~~ incorporated by reference. Such methods includes random packings, mats, cloths, bundles, twisted bundles, meshes, arrays etc.

Kindly amend the paragraph which beings on page 16, line 31 as follows:

In an aspect the present invention comprises a plurality of fibres extending in substantially the same direction. One way of packing such fibres relevant to the present invention is disclosed in US 5,690,823 hereby ~~included~~ incorporated by reference.

Kindly amend the paragraph which beings on page 16, line 35 as follows:

In many embodiments according to the present invention the membrane is porous, and ~~have~~ has pores in the range 0,001-100 micron, such as pores in the range 0,001-10 micron and preferably in the range 0,01-0,2 micron.

Kindly amend the paragraph which beings on page 17, line 26 as follows:

A In a particular preferred embodiment said tubular members comprise two separate set of hollow tubular members, both sets of said hollow tubular members comprising an inlet and an outlet plenum communicating with the outside of said pressurized vessel, and wherein two separate fluids may be contacted with the inner surface of said hollow tubular members, and wherein two different emulsions of said fluids in said first fluid contacting the outer surface of said hollow tubular members are formed.

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Kindly amend the paragraph which beings on page 17, line 33 as follows:

Many applications of the present invention involve expanding said first fluid containing said micro emulsion(s) ~~is/are expanded~~ in a device external to the pressurised vessel. Said expansion in said external device may be performed in a controlled manner within said vessel and/or it may be expanded through a nozzle into said external device by e.g. a RESS or RESOLV technique.

Kindly amend the paragraph which beings on page 18, line 1 as follows:

A number of important applications relates to deposition of the content of said micro emulsions formed on the surface of a substrate such as on the surface of solid material present in said external device.

Kindly amend the paragraph which beings on page 18, line 18 as follows:

In another embodiment according to the present invention said material being deposited ~~material~~ comprises a semi-conducting material.

Kindly amend the paragraph which beings on page 18, line 24 as follows:

Advantageously the deposited material constitutes a layer of primary particles having an average diameter of at the most 30 nm such as at the most 20 nm, such as an ave-rage diameter of at the most 10 nm.

Kindly amend the paragraph which beings on page 19, line 3 as follows:

Additionally, said treated solid material may according to the present invention comprise a fuel cell material.

Kindly amend the paragraph which beings on page 23, line 20 as follows:

In still further applications of the present invention said micro emulsions contained in said fluid stream (F) are used as templates for producing nano-scaled primary partic-les. For such embodiments said external device (9) may comprise a separation device, wherein said primary particles produced may be separated from said first fluid, second fluid and/or one or more

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surfactants. In applications, wherein a substantially dry product is desired said external separation device (9) may comprise a filter such as a bag filter or membrane filter. In other applications, wherein a product comprising said primary particles suspended in a liquid is produced, said external separation device may comprise a vessel containing said liquid for suspending said primary particles. Said liquid ~~will in~~ may in such application act as an antisolvent. Many embodiments for production of both a substantially dry particle product and a product comprising said primary particles suspended in a liquid, involve expanding said fluid stream (F) prior to, and/or into and/or within said external separation device, e.g. through a nozzle like in the RESS and RESOLV techniques.

Kindly amend the paragraph which begins on page 23, line 35 as follows:

Fig. 5 shows an example of a pressurised vessel according to an embodiment of the present invention. Said pressurised vessel comprises a plurality of hollow tubular members extending in substantially the same direction and communicating with both an inlet and an outlet plenum. The lumen side (internal surface) of said plurality of hollow tubular members may be sealed from the shell side (outer surface) by "potting" the hollow tubular members in both ends using a potting material. The potting of hollow tubular members may be carried out in any suitable manner and such procedures are well known in the art (e.g. US 3,422,008, US3,339,341, US 3,442,389, US 3,455,460, US 3,690,465, US 4,207,192, US 5,264,171, EP 0562520A1 etc.), all of which are incorporated by reference. The potting material may be organic or inorganic or a mixture thereof. Suitable potting materials are well known and described in US 4,369,605 and US 3,422,008 incorporated herein by reference. The plurality of hollow tubular members may be arranged in a pressurised vessel as shown in the figure 5a. The first fluid and surfactant(s) is typically introduced through an inlet port on the shell side of the hollow tubular members, and contacts the outer surface of said hollow tubular members during the passage between the interstices between the hollow tubular members in said hollow tubular member array as shown in the drawing. The second fluid is introduced into said inlet plenum and is distributed to the lumen side (inner surface) of said tubular member(s). At least part of said fluid permeating through the membrane walls of said tubular members so as to obtain a controlled addition of said second fluid and/or dissolved substances to said first fluid on the outer surface of said hollow tubular members. The

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hydrophilic part of the surfactant(s) combines with the micron- or nano-sized droplets of said second fluid formed at the outer surface of said membrane part of said hollow tubular members as illustrated in figure 5c-5d. The micro emulsions of said second fluid in said first fluid, which optionally contain substances dissolved and/or dispersed therein may be continuously withdrawn from the hollow tubular member containing pressurised vessel as indicated.

Kindly amend the paragraph which beings on page 26, line 26 as follows:

Said promotion of water-in-CO₂ emulsions may be performed by withdrawing part of the fluid or fluid mixture present within said pressurized vessel to a re-circulation loop comprising one or more mixing zones for promotion of said formation said water-in-CO₂ emulsions. At least one of said mixing zones in such embodiments is typically positioned within a pressurized container, and is at least partly provided by a high shear rate mixer located within said pressurized container. It should be understood that said pressurized container ~~de~~ does not necessarily have any structural difference from a pressurized vessel, but the word container is used for clarity.

Kindly amend the paragraph which beings on page 26, line 35 as follows:

In many embodiments according to the present invention said high shear rate mixing is preferably obtained by applying a rotor and stator impeller such as shown in the figures 2-3 and described above. After passing said pressurized container the fluid stream comprising said water-in-CO₂ emulsions is fed back to said pressurized vessel.

Kindly amend the paragraph which beings on page 28, line 23 as follows:

Non-limiting examples of applications for use of said solvent includes extraction or dissolution of proteins and polypeptides, metal compounds, and processes for cleaning of textiles, metal and semiconductor parts etc.

Kindly amend the paragraph which beings on page 28, line 37 as follows:

This may be performed by controlling the flow rate and inlet temperature of said second fluid, and withdrawing the remaining part from said outlet plenum for said second fluid to a an external re-circulation loop, wherein heat is added and/or extracted in the re-circulation loop and feeding

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the fluid back to the inlet plenum for said second fluid.

Kindly amend the paragraph which beings on page 29, line 11 as follows:

The fluid volume being withdrawn from said pressurized vessel to said re-circulation loop described in illustrative example 1, may often be relatively high. The fluid volume being withdrawn may correspond to exchange of at least 0,1 fluid volume within said pressurized vessel per minute, such as at least 0,25 fluid volume exchanges per minute, preferably the fluid volume being withdrawn corresponds to at least 0,5 fluid volume exchanges within said pressurized vessel per minute, and even more preferable the fluid volume being withdrawn from said pressurized vessel corresponds to at least 1 volume exchanges per minute, and advantageously the fluid volume being withdrawn from said pressurized vessel corresponds to exchange of at least 2 vessel volumes per minute such as exchange of at least 5 vessel volumes per minute.

Kindly amend the paragraph which beings on page 29, line 22 as follows:

Hence, many embodiments according to the present invention comprises the step of controlling the temperature within said pressurized vessel by controlling the temperature within said re-circulation loop.

Kindly amend the paragraph which beings on page 31, line 13 as follows:

The compressed CO₂ stream containing said micro emulsions formed may be used as a carrier to transport said micro emulsions to an external device, e.g. a vessel containing a solid material for deposition of said dissolved species or to a particle formation device. An example of an application, wherein a deposition is involved may be for production of biocatalysts by deposition of enzymes on a polymeric matrix. Another important embodiment may be the use of micro emulsions formed in processes for production of fine particles such as particles in the nano- or micrometer range as further exemplified below.

Kindly amend the paragraph which beings on page 31, line 25 as follows:

In one embodiment according to the present invention two or more emulsions are formed in the

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same pressurized vessel by applying two or more separate sets of hollow fibres. All sets of hollow fibres ~~comprising~~ comprise an inlet and an outlet plenum communicating with the outside of said hollow tubular member containing device, and wherein two or more separate fluids may be contacted with the inner surface of said hollow tubular members, thereby producing two or more different emulsions of said fluids in the first fluid contacting the outer surface of said hollow tubular members.

Kindly amend the paragraph which beings on page 32, line 24 as follows:

In both cases the exchange between the different micellar cavities may be enhanced by applying a pulsation effect, and/or an ultrasound effect and/or an vibrating surface effect in the device, wherein said exchange between different micellar cavities occurs. The pulsation effect may be performed by modulating the density of said first fluid containing said emulsions between an uppermost and a lowermost level. This density modulation may be performed by modulating the pressure and/or the temperature. Typically the density change between the uppermost and lowermost density level is up to 75 %, such as up to 50 %, and preferable up to 30 % and the density modulation may be repeated multiple times such as 5-100 times.

Kindly amend the paragraph which beings on page 32, line 37 as follows:

An important micro emulsion system according to the present invention may be the use of the micro emulsions as templates for the syntheses of particles. Many such systems involve intermicellar exchange of different dissolved and/or dispersed substances. When such micelles collides mixing and reaction between such reactants contained in the different micellar cavities is facilitated.

Kindly amend the paragraph which beings on page 33, line 5 as follows:

An example of such system may be a reverse micelle containing a metal salt dissolved in the water core and sulfide ions in the water core of another micelle. Intermicellar exchange results in the formation of metal sulfide nanoparticles within the nanoscale reverse micelle cavities. Particle growth stops due to the limitation of the particle size that the micelle can support, and the particle size may be controlled by changing the CO₂ solvent properties.

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Kindly amend the paragraph which beings on page 33, line 12 as follows:

In another preferred embodiment a compressed water stream containing said metal containing substances dissolved or dispersed therein, is introduced into said pressurized vessel, and a fluid stream comprising said micro emulsions with said dissolved or dispersed in the water core is withdrawn to an external device, wherein said fluid comprising said micro emulsions contact with a third fluid comprising a reducing agent such as an alcohol, hydrazine, hydrides, alkalides, Grignard reagents etc. is promoted. Said reducing agent may be contained within a micro emulsion contained in said third fluid. Contact between micelles containing the metal and the reducing agent may lead to formation of metal nano-particles. The formed metal nanoparticles may be harvested by expansion of the first fluid, and collection in conventional devices for particle collection, or might be deposited directly on a substrate, as a surface treatment of said substrate to form the final product.

Kindly amend the paragraph which beings on page 33, line 37 as follows:

A preferred embodiment according to the present invention is the formation of fine particles with subsequent deposition in a thin layer on a solid material comprising the end product. Nonlimiting examples of such applications is are ceramic membranes, fuel cells, solar cells, semiconductors, self cleaning mirrors, conducting polymers, catalysts etc.

Kindly amend the paragraph which beings on page 34, line 29 as follows:

As described in illustrative example 6, one of main the problems related to processing of biomolecules such as peptides and proteins e.g. enzymes etc., is that they easily ~~løse~~ lose bioactivity in contact with the organic solvent typically employed for supercritical processing of such biomolecules due to denaturation of the steric composition and they may further change crystallinity in the absence of water.

Kindly amend the paragraph which beings on page 34, line 35 as follows:

A new supercritical emulsion drying technique for preparation of inhalable protein particles was disclosed by J.Jung et al. at the 6th International Symposium on Supercritical Fluids in Versailles

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(France), April 28.-30., 2003 (J. Jung, F. Leboeuf, and M. Perrut, "Preparation of inhable protein particles by SCF Emulsion Drying", In Proc. of 6th International Symposium on Supercritical Fluids, Tome 3: Materials Processing, p.1837-1842, 2003, ISBN 2-905-267-37-02, and M. Perrut et al, FR0106403), hereby ~~included~~ incorporated herein by reference.

Kindly amend the paragraph which beings on page 35, line 5 as follows:

In this technique, water-in-oil emulsion droplets containing dissolved proteins such as trypsin, catalase, lactase and insuline etc. ~~was~~ were sprayed into a continuous feed of high pressure carbon dioxide. Protein particles precipitated as micron sized particles as a result of expansion of the droplets and removal of water by the CO₂ organic solvent mixture. The micron size particles showed a significantly higher preservation of bioactivity than for usual supercritical processing.